Creep-deformation behavior of $(\text{Mo}_{0.85}\text{Nb}_{0.15})\text{Si}_2$ lamellar-structured C40/C11_b two-phase crystals

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Abstract

The creep-deformation behavior of $(\text{Mo}_{0.85}\text{Nb}_{0.15})\text{Si}_2$ crystals that were composed of C40 and C11_b two-phases with an oriented lamellar microstructure was examined over 1200-1400 °C under compression. The creep behavior of the crystals varied greatly depending on the orientation of the loading axis with respect to the lamellar interfaces. The steady-state creep strain rate (SSCR) when the loading orientation was parallel to the lamellar interfaces (0°-orientation) was approximately 2 orders of magnitude lower than that when the loading orientation was inclined by 45° (45°-orientation). Creep tests were performed also with single crystals of C40-single-phase $(\text{Mo}_{0.85}\text{Nb}_{0.15})\text{Si}_2$, and proved that the C40-phase effectively acts as a strengthening phase in the creep behavior of the C40/C11_b duplex-phase crystals. The variant-1-type C11_b phase grains, which have a loading orientation parallel to [001], were also found to act as an effective strengthening component. The SSCR of the 0°-orientation was succeeded to be further decreased by controlling the lamellar microstructure via the addition of Cr. The SSCR of the 1 at.-%-Cr-added lamellar-structured crystal under 300 MPa in the 0°-orientation was approximately a third of that of the non-added ternary crystal.

Keywords: *transition-metal silicides; creep test; high-temperature deformation; lamella; dislocation*

1. Introduction

There is a great demand for materials that can tolerate operating temperatures greater than 1200 °C, i.e., higher than that of Ni-based superalloys. Transition-metal disilicides (TMDs) have received much attention because they are promising candidates for such ultra-high-temperature (UHT) structural materials. MoSi₂ is one of the most promising TMDs because of its high melting temperature, good oxidation resistance, relatively low density, and high thermal conductivity [1–5]. MoSi₂ crystallizes in the C11_b structure, which is based on a tetragonal unit cell. The relatively high crystal symmetry of this structure means that many slip systems, such as {110}<111] and {013}<331], can be operative [1,4]. Here, the mixed notation of {*hkl*} and <*uvw*> differentiates the first two indices from the third index, which does not play the same role as the first two because of the tetragonality of the C11_b-MoSi₂ system. Owing to the operation of those slip systems, MoSi₂ exhibits some level of plasticity even at room temperature [4,5]. However, the serious drawbacks of a low fracture toughness at low temperatures and insufficient high-temperature creep strength must be improved before MoSi₂ is used in practical applications.

We have proposed to combine different TMDs that have similar properties as a facile approach to overcome the aforementioned problems [6,7]. In particular, C40-structured NbSi₂, which has a hexagonal unit cell, is the most promising candidate for strengthening C11_b-MoSi₂ [3,6–16]. The C11_b and C40 crystal structures are closely related to each other because the {110} atomic arrangement in the tetragonal C11_b structure is almost identical to the (0001) arrangement in the hexagonal C40 structure. The structures only differ from each other in the stacking sequences of the common atomic layers, with the C11_b and C40 structures based on two-fold and three-fold stacking sequences, respectively. By focusing on the similarity of the crystal structures, we succeeded in producing a C40/C11_b duplex-phase crystal with an oriented lamellar microstructure [7]. Fig. 1(a) shows the microstructure of the resulting duplex-phase

crystal. The oriented lamellar microstructure was developed by annealing a Mo-oversaturated ($\text{Mo}_{0.85}\text{Nb}_{0.15}$) Si_2 single-crystal with the C40 structure, which was grown via the floating-zone (FZ) method. Owing to the similarity of the aforementioned crystal structures, C11_b phases were precipitated in the C40 single crystal during the annealing process so as to their common planes are aligned parallel to each other at the lamellar interfaces. Hence, the C40 and C11_b phases exhibited distinct crystallographic orientation relationships of $(0001)_{\text{C40}} // \{110\}_{\text{C11b}}$ and $\langle \bar{1}2\bar{1}0 \rangle_{\text{C40}} // \langle 1\bar{1}0 \rangle_{\text{C11b}}$. However, on the common plane, there was a slight distortion in the atomic arrangements; the [110] axis of the C11_b structure and [0001] axis of the C40 structure possess two-fold and six-fold rotational symmetries, respectively. Therefore, three variants of the C11_b phase, which were designated variants 1 (V1), 2 (V2), and 3 (V3), were formed within the C40 single-crystal, and are represented by the following orientation relationships.

$$\text{V1: } (0001)_{\text{C40}} // (110)_{\text{C11b}}, \quad [\bar{1}2\bar{1}0]_{\text{C40}} // [1\bar{1}0]_{\text{C11b}}, \quad [10\bar{1}0]_{\text{C40}} // [001]_{\text{C11b}} \quad (1)$$

$$\text{V2: } (0001)_{\text{C40}} // (110)_{\text{C11b}}, \quad [2\bar{1}\bar{1}0]_{\text{C40}} // [1\bar{1}0]_{\text{C11b}}, \quad [0\bar{1}10]_{\text{C40}} // [001]_{\text{C11b}} \quad (2)$$

$$\text{V3: } (0001)_{\text{C40}} // (110)_{\text{C11b}}, \quad [\bar{1}\bar{1}20]_{\text{C40}} // [1\bar{1}0]_{\text{C11b}}, \quad [\bar{1}100]_{\text{C40}} // [001]_{\text{C11b}} \quad (3)$$

This was actually confirmed by the selected area electron diffraction (SAED) pattern analysis in the transmission electron microscopy (TEM) observation [7], as indicated in the appendix Fig. A.1(a-c). These crystallographic orientation relationships (hereinafter referred to as variant orientation relationships) are schematically drawn in [appendix Fig. A.1\(d\)](#). The compositions of the C40 and C11_b phases in the lamellar crystal were evaluated to be approximately ($\text{Mo}_{0.79}\text{Nb}_{0.21}$) Si_2 and ($\text{Mo}_{0.94}\text{Nb}_{0.06}$) Si_2 , respectively [6].

The development of a lamellar microstructure effectively increases the room-temperature fracture toughness of ($\text{Mo}_{0.85}\text{Nb}_{0.15}$) Si_2 crystals [10,15]. In addition, C40/C11_b crystals exhibit superior high-temperature strength compared to the polycrystalline MoSi₂ [6,11]. Thus, lamellar-structured ($\text{Mo}_{0.85}\text{Nb}_{0.15}$) Si_2 crystals have a potential as a next-generation UHT structural material. It must be focused here that both the fracture toughness and high-temperature strength were found to exhibit strong

orientation dependencies with respect to the lamellar interfaces [10,11,15]. Thus, controlling the microstructure and loading orientation in the C40/C11_b duplex-phase crystals is essential for obtaining superior mechanical properties.

Regarding the control over the lamellar microstructure, we found a problem with its thermal stability. As shown in Fig. 1(a), some coarse C11_b-phase grains were developed in addition to the fine lamellar-structured C11_b grains during the long-term annealing of the ternary (Mo_{0.85}Nb_{0.15})Si₂ crystals. These coarse C11_b-phase grains do not exhibit the aforementioned variant orientation relationships, and they are thought to form as a way of releasing the residual strain accumulated at the lamellar interfaces from the formation of fine lamellae [7]. We have recently shown that the thermal instability of the fine lamellar microstructure is drastically improved by the small addition of Cr [14,16]. As shown in Fig. 1(b), the almost all of coarse C11_b grains without having the variant orientation relationship disappeared, and the Cr-added duplex-phase crystal was entirely composed of the fine lamellar microstructure. The lamellar microstructure was confirmed to be stable even after annealing for 504 h at 1400 °C [17]. The origin of the improved thermal stability of the microstructure, accompanied by the segregation of added Cr atoms on the lamellar interface, has been discussed with the help of computational analyses [18,19].

In the present study, we examined the creep properties of C40/C11_b duplex-phase crystals for the first time. The creep-deformation behavior was investigated via compressive creep tests at temperatures above 1200 °C, with the orientation dependence examined in detail. By observing the deformation microstructure, the factors controlling the creep behavior were identified. In addition, the effects of the Cr addition on the creep-deformation behavior were also examined by focusing on the influence of variation in lamellar microstructure.

2. Experimental methods

Mother alloys with chemical compositions of $(\text{Mo}_{0.85}\text{Nb}_{0.15})\text{Si}_2$ and $(\text{Mo}_{0.85}\text{Nb}_{0.15})_{0.97}\text{Cr}_{0.03}\text{Si}_2$ (the numbers are in atomic fractions; the alloys will hereinafter be referred to as ternary and Cr-added alloys, respectively) were prepared by arc-melting high-purity Mo, Nb, Si, and Cr under an Ar atmosphere. Single crystals were grown from the mother alloys with an optical floating-zone furnace (SCI-MDH-20020, Canon Machinery) at a growth rate of 2.5 mm/h under an Ar gas flow. The Laue X-ray diffraction method was used to confirm that the single crystals were composed of a C40-single-phase. The C40-phase single crystals were then annealed at 1400 °C for 168 h under an Ar atmosphere to develop the oriented lamellar microstructure, which is composed of C40 and C11_b phases as shown in Fig.1. Details of the microstructures of the ternary and Cr-added crystals can be found in our previous paper [16]. The annealed crystals will hereinafter be referred to as lamellar-structured crystals.

From the lamellar-structured C40/C11_b duplex-phase crystals, rectangular specimens with a cross-section of 2.4×2.4 mm and height of 6 mm were cut for the creep tests by electrical discharge machining. The prepared specimens had rectangular markers with a cross-section of 0.4×0.8 mm and length of 2.4 mm near both edges for precisely measuring the creep strain, as indicated in Fig. 2(a). Two loading axes were selected for the creep tests: one parallel to $[10\bar{1}0]$ and the other inclined by 45° towards $[0001]$ in the $[1\bar{2}10]$ zone of the C40-matrix phase, as indicated in Fig. 2(b). Thus, the former is parallel to the lamellar interfaces, and the latter is inclined by 45° to the lamellar interfaces; these loading orientations will hereinafter be referred to as the 0°- and 45°-orientations, respectively. The fine, lamellar-structured, C11_b-phase grains precipitated in the C40-matrix phase exhibit variant orientation relationships with respect to the C40-matrix phase, as described by Eqs. (1–3). Based on these relationships, the Schmid factors for the expected operative slip systems in the C11_b and C40 phases under compressive creep deformation were calculated as listed in appendix

Table A.1. In addition to the duplex crystals, the creep-deformation tests were conducted also with C40-single-phase ($\text{Mo}_{0.85}\text{Nb}_{0.15}\text{Si}_2$) single crystals, and the effects of the lamellar microstructure on the creep properties were determined.

The creep tests were conducted at 1200, 1300, and 1400 °C under applied compressive stresses of 50–900 MPa under an Ar atmosphere. The creep strains were measured as a function of time with the non-contact method using a digital extensometer (model ZS32, Zimmer Japan Ltd.). A strong blue light was radiated onto the back of the specimen, and then a front-facing camera with a filter that blocked all light except for blue wavelengths detected the shadow of the specimen. This allowed the change in the gauge length between the markers to be precisely measured during the creep tests. The creep tests were conducted for 15–25 h and then stopped before fracture for most of the specimens, to evaluate the SSCR and to examine the deformation microstructure. On some specimens, the creep tests were continued to 168 h to examine the creep life of the crystals.

The deformation microstructures after the creep tests were observed with optical microscopy (OM, OLYMPUS BX51M), scanning electron microscopy (SEM, JEOL JSM-6500F), and transmission electron microscopy (TEM, JEOL JEM-3010) operated at 300 kV.

3. Results

3.1. Creep-deformation behavior of the lamellar-microstructured ($\text{Mo}_{0.85}\text{Nb}_{0.15}\text{Si}_2$) ternary crystals

3.1.1. Creep-deformation behavior in the 45°-orientation

Figs. 3(a, b) indicate the creep strain vs. time curves showing how the creep-deformation behavior varies with the applied stress and temperature, respectively, in the 45°-orientation. The specimens exhibit large creep strain rates, even in the early stages of creep deformation, and these large strain rates continued during the creep test.

While the difference between the initial transient-creep regime and steady-state region is clear in the tests performed at 1200 and 1300 °C (Fig. 3(b)), it is not clear when the applied stress varies at 1400 °C (Fig. 3(a)). Figs. 3(c, d) show the corresponding creep strain rate vs. **strain** curves for Figs. 3(a, b), respectively. From the creep strain rate vs. **strain** curves, the SSCR was determined as depicted in the figures. The SSCR increases when either the applied stress increases at a fixed temperature or the temperature increases at a fixed applied stress. The precise relationships between the SSCR and the applied stress and reciprocal of the temperature were indicated in Figs. 3(e, f), respectively. From these plots, the stress exponent, n , and the apparent activation energy, Q , for creep in the 45°-orientation were calculated to be 2.50 and 381 kJ/mol, respectively. The value of n is relatively close to that reported for monolithic MoSi₂ single crystals with "soft" loading orientations of [0 15 1] ($n = 2.19$) [20], and [210] ($n \approx 2.6$) [21], in which the creep-deformation behavior was controlled by dislocation glide and climb motions [20].

To clarify the detailed creep-deformation mechanism in the 45°-orientation, the deformation microstructure in the crept specimens was observed with TEM. Fig. 4 shows the bright-field TEM images of the deformation microstructure of a crept specimen subjected to 100 MPa at 1400 °C for 24 h. The Burgers vector of the dislocations operated in creep deformation was determined by performing $g \cdot b$ contrast analysis on the TEM images with several different reflection vectors (g -vectors). Figs. 4(a–d) show the dislocations present in a variant-1-type C11_b phase grain (C11_b-V1). A large number of dislocations can be seen when observed with $g = 110$ (Fig. 4(b)). However, focusing on the dislocations labeled "1" and "2" in Fig. 4(b), the contrasts of them were both lost when observed with $g = 006$ (Fig. 4(a)), and only small number of dislocations labeled "3" were visible with $g = 006$. In addition, the dislocations "1" and "2" lost their contrasts also with $g = 10\bar{3}$ (Fig. 4(c)) or $g = 0\bar{1}\bar{3}$ (Fig. 4 (d)), respectively. The visible/invisible criteria for dislocations whose operation is expected

in C11_b-V1 are listed in appendix Table A.2. Taking this into consideration, the Burgers vectors of the dislocations "1" and "2" were determined to be parallel to [100] and [010], respectively. This analysis result demonstrates that the Burgers vectors of the dislocations predominantly operative in the C11_b-V1 grain are <100], and small number of dislocations with <110> or <111> Burgers vectors coexisted in the crept specimen, as schematically drawn in Fig. 4(e). A large number of dislocations were observed also in the C11_b-V2 and C11_b-V3 grains. As shown in Fig. 2(b), the loading orientation in the C11_b-V2 and C11_b-V3 grains is crystallographically identical. Thus, the same mechanism must control creep-deformation behavior in them. Based on the visible/invisible criteria for dislocations in C11_b-V2, listed in appendix Table A.3, the results of the g·b contrast analysis in the C11_b-V2 (Figs. 4(f–i)) is schematically drawn in Fig. 4(j). The observation result demonstrates that the operation of dislocations with the Burgers vectors parallel to [100] and [010], i.e., <100], is predominant also in the C11_b-V2 grains as labeled "1" and "2", as similar to C11_b-V1.

In the C40 phase, the existence of dislocations that have Burgers vectors parallel to $\langle 11\bar{2}0 \rangle$ was confirmed. However, the number of dislocations in the C40 phase was much less than that in the C11_b phase grains. This suggests that the C11_b phase predominantly carry the creep strain during the deformation of the duplex crystal in the 45°-orientation.

3.1.2. Creep deformation behavior in the 0°-orientation

Figs. 5(a, b) indicate the creep strain vs. time curves showing how the creep-deformation behavior varies with the applied stress and temperature, respectively, in the 0°-orientation. The results clearly demonstrate that the creep-deformation behavior is strongly orientation-dependent. When compared the creep deformation behavior under 150 MPa at 1400 °C, the creep strain at 45°-orientation reached to ~3 % after 4 h, but it was as small as ~0.06 % at 0°-orientation.

In the 0°-orientation, a short initial transient-creep region occurs for approximately 0-3 h, and then the steady-state creep region follows for most of the conditions tested. At 1400 °C, when the applied stress is larger than 300 MPa, the creep strain rate showed the further increase as the deformation proceeds, i.e., a tertiary creep region gradually appeared at less than 25 h. Under 400 MPa, the specimen was fractured just after 6 h. When the stress is lower than 150 MPa, on the other hand, the steady-state creep deformation continued for over 168 h. At present, further details on the variations in creep life with applied stress and temperature are not sufficiently clarified yet. This is the future work we must examine.

Figs. 5(c, d) show the creep strain rate vs. **strain** curves for evaluating the SSCR. Similar to the results of the 45°-orientation tests, the SSCR increases when the applied stress and/or temperature increase. **However**, compared to the creep-deformation behavior of the 45°-orientation (Fig. 3), **the value of SSCR** is approximately 2 orders of magnitude smaller in the 0°-orientation under the same applied stress. Figs. 5(e, f) show the relationships between the SSCRs and the applied stress and reciprocal of the temperature, respectively. From these plots, the n and Q values for creep in the 0°-orientation were calculated to be 3.48 and 284 kJ/mol, respectively. This n value is much higher than that calculated for the 45°-orientation, suggesting that different mechanisms control the creep-deformation behavior in the different loading orientations.

Figs. 6(a–c) show the low-magnification bright-field TEM images of the deformation microstructure of a crept specimen subjected to 300 MPa in the 0°-orientation at 1400 °C for 24 h. By the analysis of SAED patterns along the $[2\bar{1}\bar{1}0]$ and $[1\bar{1}00]$ directions indicated in appendix Fig. **A.1(a-c)**, the three $C11_b$ variants and $C40$ -matrix phase in the lamellae were identified as indicated in the Fig. 6(a). Fig. 6(a) shows the deformation microstructure observed with the reflection vector $g = 0003$ in the $C40$ phase. By taking the variant orientation relationships between the $C40$ and $C11_b$

phases shown in [appendix Fig. A.1\(d\)](#) into consideration, the reflected $g = 0003$ vector in the C40 phase corresponds to the $g = 110$ vector in all three C11_b variants. Operation of dislocations was observed in almost all the grains (lamella). However, it should be noted that in the C11_b-V1 grains, in which the loading axis is parallel to [001], the number of dislocations is much less than that in the other C11_b variants. [Appendix Table A.1](#) shows that the Schmid factors for slip are all negligibly small except for the {013}<331> slip in the C11_b-V1 grains. Here, Ito et al. [5] reported that the operation of {013}<331> dislocations exhibits a strong yield stress anomaly (YSA) with a peak at around 1200 °C. The low number of dislocations observed in the C11_b-V1 must be ascribed to this YSA.

On the other hand, a relatively large number of dislocations are present in the C11_b-V2 and C11_b-V3 grains. To identify the Burgers vector of these dislocations, further TEM observations were conducted with other g -vectors. Fig. 6(b) shows the TEM images observed with $g = \bar{2} \bar{2} 40$ in the C40 phase. From geometric considerations, $g = \bar{2} \bar{2} 40$ corresponds to $g = \bar{1} 1 \bar{6}$ in C11_b-V1, $g = \bar{1} 1 6$ in C11_b-V2, and $g = 2 \bar{2} 0$ in C11_b-V3 grains. Most of dislocations were visible in the C11_b-V2 and C11_b-V3 grains as the same as those observed in Fig. 6(a) with $g = 110$. However, as shown in Fig. 6(c), when observed with $g = 02 \bar{2} 0$, the contrast of almost all of dislocations has been lost in the C11_b-V2, although the contrast of dislocations is still clearly visible in the C11_b-V3. The $g = 02 \bar{2} 0$ vector in the C40 phase corresponds to $g = \bar{1} \bar{1} 2$ in the C11_b-V1, $g = 00 \bar{4}$ in the C11_b-V2, and $g = \bar{1} 1 2$ in the C11_b-V3 grains. According to the visible/invisible criteria of dislocations listed in [appendix Table A.3](#), this observation result demonstrates that the Burgers vector of the dislocation predominantly operative in the C11_b-V2 grains is parallel to <100>. From the TEM observations with the other g -vectors, the same conclusion was drawn for the C11_b-V3 grains.

A relatively large number of dislocations are present also in the C40 phase. Figs.

6(d-f) show the enlargement images of dislocations in the C40 phase observed with various g -vectors. Most of dislocations labeled as "1" in the figures are strongly contrasting when observed with $g = \bar{2}\bar{2}40$ and $g = 02\bar{2}0$, as shown in Figs. 6(e) and (f). While the contrast of them is lost or it exhibits so-called "residual contrast" when observed with $g = 0003$ (Fig. 6(d)). This indicates that the Burgers vectors of most of the dislocations operated in the C40 phase do not have a c -component. By the tilting observation, the slip plane of those dislocations was confirmed to be the basal plane. Thus, they were identified as $(0001)\langle 11\bar{2}0 \rangle$ basal dislocations. A slight deviation of the loading axis from $[10\bar{1}0]$ and/or a slight crystal rotation during the creep deformation may activate their operation. However, from a geometric viewpoint, their motion cannot carry the creep strain along the loading axis. Thus, the effect of basal slips on the creep behavior of the duplex-phase crystal in the 0° -orientation must be limited.

In addition to the $\langle 11\bar{2}0 \rangle$ basal dislocations, a small number of dislocations with a c -component are also present in the C40 phase, as labeled "2" in Fig. 6(d). They are visible with $g = 0003$, but lose their contrasts with $g = \bar{2}\bar{2}40$ and $g = 02\bar{2}0$, suggesting that the Burgers vector of them is parallel to $[0001]$. The nature of dislocations which contains the c -component in their Burgers vector, is few reported in the C40 phase. As a related study, Nakano et al. [22] reported the existence of $\langle 11\bar{2}X \rangle$ -type dislocations in C40-NbSi₂ crystals deformed at 1000 °C. These non-basal dislocations may play an important role in the deformation of lamellar-structured crystals in the 0° -orientation, although the details of such a mechanism have not yet been sufficiently clarified.

3.2. Creep-deformation behavior of C40-single-phase ($Mo_{0.85}Nb_{0.15}$)Si₂ single crystals

As described in section 3.1, the lamellar-structured C40/C11_b duplex-phase crystals exhibit strongly orientation-dependent creep behavior. To discuss the origin of this

dependence, the anisotropy of creep behavior of the constituent C40 and C11_b phases themselves must be understood. Inui et al. [20] investigated the orientation dependence of the creep-deformation behavior of single-crystal C11_b-MoSi₂. However, information on the creep behavior of the C40 phase is still limited. Thus, we studied the creep-deformation behavior of the as-FZ-grown (Mo_{0.85}Nb_{0.15})Si₂ single crystals with the C40 structure. Figs. 7(a, b) show typical creep strain vs. time curves of the C40 phase in the 0°- and 45°-orientations, respectively. In addition, Figs. 7(c, d) show the corresponding creep strain rate vs. **strain** curves for evaluating the SSCR. Similar to the lamellar-structured crystals, the creep behavior of the C40-single-phase crystals is strong orientation-dependent. The SSCR measured in the 0°-orientation is approximately 2 orders of magnitude lower than that in the 45°-orientation. Focusing on the absolute values of the SSCRs, they are extremely small compared to that of the lamellar-structured crystals in both the 0°- and 45°-orientations, even though the bulk compositions of the single-phase and lamellar-structured crystals are identical. The SSCR at 1400 °C in the 45°-orientation is $4.42 \times 10^{-8} \text{ s}^{-1}$ at 250 MPa, while the SSCR in the 0°-orientation is $4.78 \times 10^{-9} \text{ s}^{-1}$ at 600 MPa. Both values are more than 2 orders of magnitude lower than that of the duplex-phase crystals. In particular, the creep strain of the single-crystals in the 0°-orientation was too low to be measured below 500 MPa at 1400 °C. This clearly demonstrates the C40 phase effectively acts as the strengthening phase for the creep deformation of the C40/C11_b lamellar-structured crystals.

It should be noticed that in the creep strain rate vs. **strain** curve of the C40 single crystals at 45°-orientations (Fig. 7(c)), the steady state region was very narrow and the strain rate was drastically increased in early stage of creep deformation, different from those measured in lamellar crystals. The time at which the creep rate drastically increases had a relatively large scatter for each specimen, but it occurred at approximately 20 h in the 45°-orientation under 250 MPa, while it occurred at around 60 h in the 0°-orientation under 600 MPa. That is, the drastic increase in the strain rate

occurred much faster in the 45°-orientation, even though the applied stress was smaller than that used in the 0°-orientation. By observing the deformation microstructure in the specimens, this drastic increase in the strain rate is ascribed to the dynamic development of a lamellar microstructure in the C40-single-phase crystals during the creep tests. Figs. 7(e, f) show the microstructure of crept specimens subjected to 600 MPa in the 0°-orientation at 1400 °C for 20 and 70 h, respectively. The lamellae developed during the creep test for 20 h (Fig. 7(e)) are fine and have a morphology similar to that reported for specimen that were statically annealed for 24 h at 1400 °C [14]. In the 0°-orientation, a change in the creep strain rate was little detected at around 20 h, even though the lamellae have started to form. In contrast, the creep strain rate drastically increased in the 45°-orientation at approximately 20 h as shown in Fig. 7(a). This indicates that the formation and development of the lamellar microstructure is greatly affected to the creep-deformation behavior in the 45°-orientation compared to that in the 0°-orientation.

However, after the creep tests were performed for 70 h, drastic increase in creep strain rate followed by fracture occurred even in the 0°-orientation. In the fractured specimen, in addition to the fine lamellae, large C11_b-phase grains that lack the flat lamellar interface have developed as indicated by the arrows in Fig. 7(f). This is similar to that observed in the specimen statically annealed for 168 h at 1400 °C (Fig. 1(a)). This result implies that although the formation of a lamellar microstructure is less affected to the creep behavior in the 0°-orientation, the development of coarse C11_b-phase grains drastically reduces the creep strength of the crystals, even in the 0°-orientation.

Figs. 8(a) and (b) show the relationships between the SSCRs of the C40-single-phase crystals and the applied stress at 1400 °C in the 45°- and 0°-orientations, respectively. In addition, Figs. 8(c) and (d) show the relationships between the SSCRs and the reciprocal of the temperature in the 45°- and 0°-orientations,

respectively. From the graphs n and Q values for the C40 phase were evaluated to be approximately 3.10 and 946 kJ/mol in the 45°-orientation, and 7.89 and 1390 kJ/mol in the 0°-orientation, respectively. Both the n and Q values are extremely high compared to that evaluated for the lamellar-structured crystals.

3.3. Variations in the creep-deformation behavior of the lamellar-structured crystals by the addition of Cr

As described in section 1, Fig. 1(b) shows that the minute addition of Cr drastically improves the thermal stability of the lamellar microstructure, via the segregation of it on the lamellar interfaces [14,16–19]. The effects of such a variation in the microstructure on the creep deformation behavior were also investigated in this study. Figs. 9(a, b) show the typical strain-time curves of the Cr-added lamellar-structured crystals in the 45°- and 0°-orientations, and Fig. 9(c, d) show the corresponding creep strain rate vs. **strain** curves. In the graphs, creep behaviors of the lamellar-structured ternary crystals subjected to the same conditions are included for comparison purposes. These curves show that the effects of the Cr addition on the creep behavior produced very different results in the 0°- and 45°-orientations. In the 0°-orientation, the SSCR is significantly reduced by the Cr addition, as shown in Fig. 9(b, d). The SSCR of the Cr-added specimen in the 0°-orientation at 1400 °C was $5.97 \times 10^{-8} \text{ s}^{-1}$, while that of the lamellar-structured ternary ($\text{Mo}_{0.85}\text{Nb}_{0.15}$) Si_2 crystal was $1.59 \times 10^{-7} \text{ s}^{-1}$. That is, the SSCR of the Cr-added crystal in the 0°-orientation was approximately a third of that of the non-added ternary crystal. However, in the 45°-orientation, the SSCR was adversely affected (increased) by the Cr addition. The SSCR under 100 MPa at 1400 °C were estimated to be $2.92 \times 10^{-6} \text{ s}^{-1}$ for the Cr-added crystal, which is more than 4-times larger than that of the ternary crystal.

4. Discussion

4.1. Origin of the anisotropic creep-deformation behavior of the duplex-phase crystal

The results of this study have shown that the creep-deformation behavior of C40/C11_b lamellar-structured crystals is strongly dependent on the loading orientation and affected by Cr addition. To summarize, Fig. 10 shows the SSCRs of the lamellar- and non-lamellar-structured C40-single-phase (Mo_{0.85}Nb_{0.15})Si₂ crystals, and Cr-added lamellar-structured crystals in the 0°- and 45°-orientations as functions of the applied stress at 1400 °C. The SSCR of binary MoSi₂ single crystals in the [001] “hard” orientation, which was reported by Inui et al. [20], is also plotted for comparison purposes. The SSCR in the 0°-orientation is significantly lower than that in the 45°-orientation for all the crystals investigated. This tendency corresponds well with the previously reported orientation dependence of the yield stress of the C40/C11_b lamellar-structured crystals [11]. In the compression tests at 1400 °C and a strain rate of $1.67 \times 10^{-4} \text{ s}^{-1}$, the yield (fracture) stress of the lamellar-structured crystals in the 0°-orientation was as high as ~890 MPa, which is approximately three times larger than that in the 45°-orientation (~270 MPa) [11]. The applied stresses employed in the present creep tests in the 0°- and 45°-orientations are not significantly different when normalized with their yield stresses; they correspond 15–45% (137–400 MPa) and 19–56% (50–150 MPa) of the yield stresses in the 0°- and 45°-orientations, respectively. Thus, the large differences between the SSCRs of the two orientations must originate from the different deformation mechanisms. Two main factors are considered for the origins of the strong orientation dependence of the creep-deformation behavior: the anisotropic creep-deformation behavior of the constituent C11_b and C40 phases (intrinsic) and the specific lamellar microstructure of the C40/C11_b duplex-phase crystals (extrinsic).

The lamellar microstructure means that in the 0°-oriented specimen, both the C40 and C11_b phases must be plastically deformed for the specimen to be macroscopically deformed. Contrastingly, in the ideal case for the 45°-oriented specimen, only the

deformation of either the C40 or C11_b phase can induce macroscopic deformation if the specimen is entirely composed of a perfect, plate-like, lamellar microstructure, as schematically drawn in Fig. 10.

Regarding the intrinsic factor, we first clarified in section 3.2 that the creep behavior of the C40 phase is strong orientation-dependent. This is ascribed to the limitations of the operative slip systems in the C40 phase. The operative deformation mode in C40-NbSi₂ crystals has been investigated by performing compression tests on single crystals [22–24]. The results of these studies showed that only the (0001)<11 $\bar{2}$ 0> basal slip was the predominant operative slip system, which was independent of temperature. This basal slip system was also confirmed in our previous study on C40-structured (Mo_{0.85}Nb_{0.15})Si₂ single crystals [11]. The Schmid factor for the (0001) basal slip is negligibly small in the 0°-oriented crystal since the basal plane is parallel to the loading axis, while it exhibits a large value (0.433) in the 45°-oriented crystal (appendix Table A.1). This must be the origin of the strong orientation-dependent creep behavior of the C40-single-phase single crystals, as shown in Fig. 7, and this anisotropy must strongly affect the creep deformation behavior of the lamellar crystals.

It must be noted here, however, that the *n* values for the creep deformation of the C40-single-phase specimens are 3.10 in the 45°-orientation and 7.89 at 0°-orientation, which are very different from that of the lamellar-structured crystals (2.50 and 3.48, respectively), even though the alloy compositions are identical. This is a surprising fact, and it obviously demonstrates that the creep deformation behavior of the lamellar-structured crystals is not governed only by the C40 phase but it significantly affected by the C11_b phases. Contrary to the C40 phase, five slip systems are reported to be operative in C11_b-MoSi₂ [4,5], as shown in appendix Table A.1. It was reported that MoSi₂ single crystals also exhibit orientation-dependent creep behavior via the change in operative slip systems [20]. A relatively large creep strain was measured in binary MoSi₂ for creep in the [0 15 1] orientation because of the easy operation of the

{110}<111] slip, while a much smaller SSCR was measured in the [001] orientation, where only the {013}<331] slip is expected to operate. The {013}<331] slip is known to exhibit a YSA at high temperatures (see section 3.1.2) [5]. Thus, the critical resolved shear stress (CRSS) of the {013} slip at 1400 °C can be as high as ~580 MPa. This caused the significantly reduced SSCR in the [001] orientation [20]. Such differences in the operative slip systems and CRSS values cause the anisotropic creep-deformation behavior of MoSi₂.

Based on these plastic anisotropies of the constituent phases, the creep-deformation mechanisms of the lamellar-structured C40/C11_b crystals in the 45°- and 0°-orientations are discussed in the following sections, including the effects of the lamellar microstructure.

4.2. Creep-deformation mechanism in the 45°-orientation

The lamellar-structured C11_b grains in the C40/C11_b crystals have variant orientation relationships with respect to the C40-matrix phase, as indicated in [appendix Fig. A.1\(d\)](#). Based on these relationships, the temperature dependencies of the yield stresses for the three C11_b variants and C40 phase in duplex-crystals deformed in the 45°-orientation were virtually estimated as shown in Fig. 11(a), which was partly reported in our previous paper [11]. For these calculations, the CRSS of the operative deformation modes in the C11_b variants were estimated from the literature data reported for MoSi₂ single crystals [4,5], for simplicity. In addition, the CRSS value for the C40 phase was newly evaluated in this study by using the C40-single-phase (Mo_{0.80}Nb_{0.20})Si₂ single crystals, which have a similar composition to that of the matrix phase in the duplex-phase crystal. According to the estimations shown in Fig. 11(a), the predominantly operative dislocations predicted for the C11_b-V1 grain has a Burgers vector parallel to <100], while that for both the C11_b-V2 and C11_b-V3 phases have Burgers vectors parallel to <100] and <111] in the 45°-orientation at 1400 °C. Here, the

addition of Nb to MoSi₂ was previously reported to largely increase the CRSS for {110}<111> slip because of the enhancement of YSA [20]. Thus, the motion of the <100> dislocation is expected to be the predominant operative dislocation in all three of the C11_b variants. This is in good agreement with the TEM images of the crept specimen shown in Fig. 4. In the C40 phase, the most likely operative slip system is the (0001)<11 $\bar{2}$ 0> basal slip, but its CRSS is much higher than that of the <100> dislocations in the C11_b variants, which could be confirmed in Fig. 11(a). As the origin of the high CRSS for the basal slip in (Mo_{0.80}Nb_{0.20})Si₂, it is proposed by the high-resolution TEM observation results that the dissociation mode of 1/3<11 $\bar{2}$ 0> basal dislocations into 1/6<11 $\bar{2}$ 0> superpartials in C40-NbSi₂ changes from the ordinary coplanar-type to the three-dimensional complicated shyncroshear-type as increasing in the Mo content [25].

As discussed in section 4.1, the 45°-oriented specimen can only be macroscopically deformed by the deformation of either the C11_b phase or C40 phase, but not both, if the lamellae are ideally aligned.

Therefore, it is plausible that the operation of the <100> dislocations in the C11_b phase predominantly controls the creep behavior of the lamellar-structured crystals in the 45°-orientation. While there is no literature reporting the creep deformation of MoSi₂ via the motion of <100> dislocations, Boss [21] reported the creep-deformation behavior of MoSi₂ single crystals in the [210] orientation. Although the paper did not describe the operative slip system for the loading orientation, the operations of {011}<100> and {023}<100> slips are expected in the [210] loading orientation when the Schmid factor and CRSS are considered. In the creep test in the [210] orientation at 1200 °C, n was estimated to be ~2.6 from the graph reported by Boss [21]. This value is close to that obtained in the present study for the 45°-orientation (n = 2.50). In contrast, the n value of the C40-single-phase crystal in the 45°-orientation was evaluated to be 3.10 (Fig. 8(a)), which is much higher than that in the duplex-phase crystal. This

supports our conclusion that the $\langle 100 \rangle$ slip in MoSi_2 predominantly controls the creep behavior of the duplex-phase crystals in the 45° -orientation.

The slight inconsistencies in the n values obtained in the present study and Boss's study may originate from imperfections in the lamellar microstructure of the ternary $(\text{Mo}_{0.85}\text{Nb}_{0.15})\text{Si}_2$ crystals. Fig. 1(a) shows that not all of the C11_b phase had a fine lamellar microstructure in the ternary crystal; some coarse C11_b grains had also developed. Thus, for the macroscopic deformation of specimens in the 45° -orientation, the activation of other slip systems, such as $\{110\}\langle 111 \rangle$ slip, may be partly required in addition to the $\langle 100 \rangle$ slip for such coarse grains. Indeed, the compressive yield stress of the ternary lamellar-structured crystals in the 45° -orientation at 1400°C (Fig. 11(a)) is somewhat higher than the ideal values expected for only the deformation of C11_b variants. This must cause the n values to deviate from that of MoSi_2 crept by $\langle 100 \rangle$ dislocations.

4.3. Creep-deformation mechanism in the 0° -orientation

Fig. 11(b) shows the expected temperature dependencies of the yield stresses for the three C11_b variants and C40 phase in the lamellar-structured crystals deformed in the 0° -orientation. One difference from the creep-deformation behavior in the 45° -orientation is that the deformation of both the C40 and C11_b phases must be required for the creep behavior of the duplex-phase crystals in the 0° -orientation. This is because the shear deformation must occur beyond the lamellar interfaces of the specimens in the 0° -orientation to be macroscopically deformed. As clarified in section 3.2, the C40 phase effectively acts as the strengthening phase in the 0° -orientation because the operation of the (0001) basal slip is prohibited owing to its negligible Schmid factor, and in addition its relatively high CRSS owing to the shynicroshear-type dissociation of $\langle 11\bar{2}0 \rangle$ basal dislocations [25]. Actually, the yield (fracture) stress of the $(\text{Mo}_{0.80}\text{Nb}_{0.20})\text{Si}_2$ C40 -single-phase crystals that corresponds to the matrix phase in

the lamellar-structured crystal exhibit high values at 1400 °C in the 0°-orientation (approximately 1270 MPa, see Fig. 11(b)). Regarding the C11_b phase, the 0°-orientation corresponds to [001] in the C11_b-V1 grains (Fig. 2(b)). As described in section 4.1 and Fig. 10, the [001] axis is the strongest direction in C11_b-MoSi₂ because only the {013} slip that shows a strong YSA can operate in this orientation. Thus, the expected yield stress of the C11_b-V1 phase exhibits extremely high values that are comparable to that of the C40 phase, as shown in Fig. 11(b). Therefore, the C11_b-V1 grains must act as an effective reinforcing component, as well as the C40 phase, in the 0°-orientation, while the deformation of the C11_b-V2 and C11_b-V3 grains by the operation of <100> dislocations is expected to be relatively easy. These hypotheses are in good agreement with the TEM images shown in Fig. 6.

Thus, the reason why the creep strains obtained in the 0°-orientation were much smaller than that in the 45°-orientation is ascribed to the existence of the C40 and C11_b-V1 grains that act as effective strengthening phases. That is, the creep behavior in the 0°-orientation is governed by the deformation of these strengthening phases. In reality, the yield (fracture) stress of the duplex-phase crystals in the 0°-orientation at 1400 °C showed the high value of approximately 890 MPa, which is close to that of C40 and C11_b-V1 grains (Fig. 11(b)).

The *n* value of the duplex-phase crystal in the 0°-orientation was estimated to be 3.48, which is between that of the C40 phase (*n* = 7.89) and that of the C11_b-MoSi₂ (*n* = 2.43, 2.19, and 2.6 along [001], [0 15 1], and [210], respectively) [20,21]. Regarding the evaluation of the creep-deformation behavior of the two-phase alloy, several analytical methods have been proposed, including the models based on the rule of mixture with the assumption of isostrain rate by McDanel et al. [26], shear-lag theory by Kelly and Street [27], and the self-consistent theory with the Eshleby's classical analysis by Chen and Argon [28]. Currently, consideration must be given to which model should be used to analyze the present duplex-phase crystals. However, for all of the models, the value

of n for the two-phase crystal should have the intermediate value between that of the constituent phases. This is in good agreement with the results of this study, demonstrating the validity of the experiments.

As another important approach to comprehend the creep deformation of lamellar-structured C40/C11_b duplex crystal, to refer the previous reports on the creep deformation behavior of various directionally solidified (DS) eutectic multi-phase alloys [29-33] must be useful. Among them, Mason and Van Aken [33] reported that the creep deformation behavior of DS MoSi₂/Mo₅Si₃ crystal with aligned microstructure can be described by the following equation, on the basis of the discussion given by Schmidt-Whitley in DS Al-CuAl₂ [29];

$$\dot{\epsilon} = A (\sigma^n)(\lambda^m)\exp(-Q/RT) \quad (4)$$

where $\dot{\epsilon}$ is the creep strain rate, A is a rate constant, σ is the stress, n is the stress exponent, λ is the average lamellar spacing, m is the lamellar spacing exponent, Q is the activation energy, R is the universal gas constant, and T is the absolute temperature. In the DS MoSi₂/Mo₅Si₃ crystal, they reported the values of $A = 1.4 \times 10^{-4}$, $n = 4.1$, $m = 1.2$, and $Q = 355$ kJ/mol, respectively [33]. On the lamellar spacing exponent m , theoretically it should be equal to 1 for a perfectly aligned and continuous fiber (or plate) reinforced composite, but it will be as high as 3 if fiber (or plate) rupture occurs [33]. The variation in creep deformation behavior with lamellar spacing has not been clarified in the C40/C11_b duplex crystal, and thus the spacing exponent m is not determined yet. The determination of this must be important for understanding further details of the controlling factors of the creep deformation behavior of C40/C11_b duplex crystal.

On the basis of equ. (4), at the present state the relation between σ and $\dot{\epsilon} \cdot \exp(Q/RT)$ was examined for C40/C11_b duplex crystal as indicated in Fig. 12. By a linear

regression analysis for all of the data obtained in this study to equ. (4), the best fit of the activation energy Q were estimated to be 386 kJ/mol with $n = 2.51$ at 45° -orientation, and 285 kJ/mol with $n = 3.43$ at 0° -orientation, respectively. These values are in good agreement with the results obtained using the Arrhenius relationship shown in Fig. 3(e, f) and Fig. 5(e, f). This strengthens the reliability of the n and Q values evaluated in this study.

The evaluated Q value of the duplex-phase crystal in the 0° -orientation in Fig. 5(f), $Q = 284$ kJ/mol, is close to that of MoSi_2 along $[001]$ (327 kJ/mol). The Q values for the diffusion of Si and Mo in MoSi_2 were reported to be 186–225 kJ/mol and 468–586 kJ/mol, respectively [34]. The Q value for Mo diffusion in MoSi_2 is much higher than the creep activation energy obtained in the present study. Thus, at the present moment it is considered that the creep deformation of the duplex-phase crystal is not simply controlled by the lattice diffusion of transition metals, but it may be predominantly controlled by dislocation glide and climb, which is similar to the creep deformation in MoSi_2 single crystals [20]. However, further analyses are required to conclude this. Particularly, as described above, the determination of the lamellar spacing exponent, m , in equ. (4) is strongly required to clarify the influence of diffusional relaxation along the interfaces etc. on the creep-deformation behavior [33, 35].

4.4. Cr-induced hardening and softening mechanisms of the creep-deformation behaviors

By adding minute amounts of Cr, the creep behavior of the lamellar-structured crystals was drastically altered. For one of the reasons, the Cr addition might affect the intrinsic creep properties of the constituent C11_b and C40 phases. The SSCR of MoSi_2 is reduced by the addition of Re, Nb, etc. [20], although the effect of Cr addition has not yet been sufficiently clarified for both the C11_b and C40 phases. However, in the C40/C11_b duplex-phase crystals, the variation in the lamellar microstructure must be the

predominant effect on the creep-deformation behavior. In the 45°-orientation, the Cr addition greatly increased the SSCR. This must be because the Cr aided the development of a well-aligned lamellar microstructure. Fig. 1(b) shows that coarse C11_b grains present in the ternary (Mo_{0.85}Nb_{0.15})Si₂ crystal have almost disappeared in the Cr-added crystal. In the Cr-added crystals of the present study, most of the C11_b phases exhibited fine plate-like shapes and a perfectly aligned lamellar microstructure was achieved. Many of the plate-like C11_b-phase grains ran the entire length of the specimens, unlike those in the ternary crystals. Thus, the {011}<100] slip and other slip systems can easily operate in the C11_b phases without being frequently blocked by grain boundaries. This must be responsible for the large increase in the SSCR.

Contrastingly, for the creep tests in the 0°-orientation, the SSCR was greatly reduced by the Cr addition. To explain this, Figs. 13(a) and (c) show crystal orientation maps of the C11_b phases in the ternary and Cr-added lamellar-structured crystals, created by analyzing the backscattered electron diffraction pattern in the SEM (SEM-EBSD). The red, pink, and purple grains in the maps represent the C11_b-V1, V2, and V3 grains, respectively. The analysis results clearly demonstrates that the volume fraction of coarse C11_b phase grains without a variant orientation relationship is drastically reduced by the Cr addition. The Cr-added crystal was almost entirely composed of the C40 phase and C11_b variant grains. In the deformation of the lamellar-structured crystal in the 0°-orientation, the C40 phase and C11_b-V1 grains with a [001] loading axis can be regarded as the strengthening phase, while the C11_b-V2 and C11_b-V3 grains and coarse, randomly oriented, C11_b grains can be considered the soft phase, as discussed in the section 4.3. Regarding this viewpoint, Figs. 13(b) and (d) show the differences in volume fractions of the constituent phases in the ternary and Cr-added lamellar-structured crystals; the gray segments represent the strengthening phase. The decreased volume fraction of the coarse C11_b-phase grains in the Cr-added crystal increases the volume fraction of C11_b-V1 grains. In addition, the volume

fraction of the C40 phase is also increased by the Cr addition because of the increase in its phase stability [24]. This is why the addition of Cr effectively improves the creep strength of the lamellar-structured crystals in the 0°-orientation. As a result, superior creep resistance of the Cr-added, lamellar-structured crystals to that of MoSi₂ crystals with the strongest [001] orientation could be successfully achieved. However, it was found that the Cr addition simultaneously enhanced the anisotropic creep behavior, while softening the creep resistance in the 45°-orientation. This strong anisotropy must be considered when evaluating the future practical use of such crystals.

5. Conclusions

The creep-deformation behavior of (Mo_{0.85}Nb_{0.15})Si₂ crystals with a C40/C11_b lamellar microstructure was examined. The effects of Cr addition on the creep deformation behavior of the lamellar-structured crystals was also investigated. The key results are summarized below.

- (1) The creep-deformation behavior of the lamellar-structured crystal was strong orientation-dependent. The SSCR in the 0°-orientation was approximately 2 orders of magnitude lower than that in the 45°-orientation.
- (2) In the 45°-orientation, the deformation of the C11_b phase grains by the operation of <100] dislocations predominantly controlled the creep-deformation behavior of the lamellar-structured crystal. While in the 0°-orientation, both the C40 and C11_b phases contribute to the creep deformation. However, the C40 phase exhibited a high yield strength because the Schmid factor for the dominant basal slip is negligibly small, in addition to its high CRSS. In addition, the C11_b phase with a [001] orientation also acted as an effective strengthening phase. The existence of these strengthening phases effectively reduced the SSCR of the lamellar-structured crystal compared to that in the 45°-orientation.
- (3) Cr addition caused the SSCR in the 0°-orientation to greatly decrease. However, it

simultaneously increased the SSCR in the 45°-orientation. That is, the anisotropy of the creep behavior was enhanced by the Cr addition because it aided the development of a perfectly aligned lamellar microstructure.

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Appendix

Some figures and tables are displayed, to complement the results described in the manuscript as follows.

*Appendix Fig. A1.

*Appendix Table A1, A2, A3.

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Figure1

Fig. 1. OM images of the microstructures of FZ-grown crystals after being annealed at 1400 °C for 168 h. (a) $(\text{Mo}_{0.85}\text{Nb}_{0.15})\text{Si}_2$ and (b) 1 at.% Cr-added crystals.

Fig. 2. (a) The shape and orientation of the specimens prepared for the creep tests. (b) Stereographic projection of the C40 and three variant C11_b phases showing the loading axes studied.

Figure3

Fig. 3. (a, b) Compressive creep-deformation curves of the lamellar-structured $(\text{Mo}_{0.85}\text{Nb}_{0.15})\text{Si}_2$ crystals crept in the 45° -orientation: (a) at 1400°C under 50, 75, 100, and 150 MPa; and (b) under 100 MPa at 1200, 1300, and 1400°C . (c, d) Corresponding creep strain rate vs. strain curves for Figs. 3(a, b), respectively. (e, f) Variations in SSCR in the 45° -orientation: (e) at 1400°C as a function of the applied stress, and (f) under 100 MPa as a function of the reciprocal of the temperature.

Figure4

Fig. 4. Bright-field TEM images of the deformation microstructure crept in the 45°-orientation at 1400 °C under 100 MPa for 24 h (to ~4.6% plastic strain). (a–d) Dislocation structure in the C11_b-V1 phase grain observed with: (a) $g = 006$, beam direction (B) $\parallel [110]$; (b) $g = 110$, B $\parallel [110]$; (c) $g = 10\bar{3}$, B $\parallel [3\bar{3}\bar{1}]$; and (d) $g = 0\bar{1}\bar{3}$, B $\parallel [3\bar{3}\bar{1}]$. (f–i) Dislocation structure in the C11_b-V2 phase grain observed with: (f) $g = \bar{1}01$, B $\parallel [1\bar{1}1]$; (g) $g = 0\bar{1}\bar{1}$, B $\parallel [1\bar{1}1]$; (h) $g = 110$, B $\parallel [1\bar{1}1]$; and (i) $g = 1\bar{1}0$, B $\parallel [001]$. (e, j) Schematic drawings showing the Burgers vectors of dislocations observed in C11_b-V1 and C11_b-V2 grains in the dotted rectangle regions, respectively.

Fig. 5. (a, b) Compressive creep-deformation curves of the lamellar-structured $(\text{Mo}_{0.85}\text{Nb}_{0.15})\text{Si}_2$ crystals crept in the 0° -orientation: (a) at 1400°C under 150, 200, 300, and 400 MPa; and (b) under 300 MPa at 1200, 1300, and 1400°C . (c, d) Corresponding creep strain rate vs. strain curves for Figs. 5(a, b), respectively. (e, f) Variations in SSCR in the 0° -orientation: (e) at 1400°C as a function of the applied stress, and (f) under 300 MPa as a function of the reciprocal of the temperature.

Fig. 6. Bright-field TEM images of the deformation microstructure crept at 1400 °C under 300 MPa in the 0°-orientation for 24 h (to ~1.5% plastic strain). (a, d) $g = 0003$ in the C40 phase, which corresponds to $g = 110$ in all three of the C11_b variants. $B \parallel [2\bar{1}\bar{1}0]_{C40}$. (b, e) $g = \bar{2}\bar{2}40$ in the C40 phase, which corresponds to $g = \bar{1}\bar{1}\bar{6}$ in C11_b-V1, $g = \bar{1}\bar{1}\bar{6}$ in C11_b-V2, and $g = 2\bar{2}0$ in C11_b-V3. $B \parallel [1\bar{1}00]_{C40}$. (c, f) $g = 02\bar{2}0$ in the C40 phase, which corresponds to $g = 1\bar{1}\bar{2}$ in C11_b-V1, $g = 00\bar{4}$ in C11_b-V2, and $g = \bar{1}\bar{1}\bar{2}$ in C11_b-V3. $B \parallel [2\bar{1}\bar{1}0]_{C40}$. (d-f) Enlargement images of dislocations in the C40 phase observed with various g -vectors, in the dotted rectangle region shown in Fig. 6(a).

Figure 7

Fig. 7. (a, b) Typical compressive creep-deformation curves of the C40-single-phase ($\text{Mo}_{0.85}\text{Nb}_{0.15}\text{Si}_2$) crystals: (a) crept at 1400 °C in the 45°-orientation under 250 MPa; and (b) crept at 1400 °C in the 0°-orientation under 600 MPa. (c, d) Corresponding creep strain rate vs. strain curves for Figs. 7(a, b), respectively. (e, f) Variation in the microstructure after creep testing the C40-single-phase crystal at 1400 °C in the 0°-orientation under 600 MPa: crept for (a) 20 h and (b) 70 h.

Figure8

Fig. 8. (a, b) Relationships between the SSCRs and the applied stress for the creep deformation of C40-single-phase $(\text{Mo}_{0.85}\text{Nb}_{0.15})\text{Si}_2$ crystals at 1400 °C: (a) in the 45°-orientation and (b) in the 0°-orientation. (c, d) Relationships between the SSCRs and the reciprocal of the temperature for the creep deformation of C40-single-phase $(\text{Mo}_{0.85}\text{Nb}_{0.15})\text{Si}_2$ crystals: (c) under 250 MPa in the 45°-orientation and (d) under 900 MPa in the 0°-orientation.

Figure9

Fig. 9. Compressive creep curves of the lamellar-structured Cr-added crystals crept at 1400 °C: (a) in the 45°-orientation under 100 MPa, and (b) in the 0°-orientation under 300 MPa. (c, d) Corresponding creep strain rate vs. strain curves for Figs. 9(a, b), respectively. For comparison purposes, the creep behaviors of the ternary ($Mo_{0.85}Nb_{0.15}$)Si₂ lamellar-structured crystal under the same test conditions are included.

Figure10

Fig. 10. Summary of the SSCRs at 1400 °C for the non-added and Cr-added lamellar-structured ($\text{Mo}_{0.85}\text{Nb}_{0.15}$) Si_2 crystals, and the non-lamellar-structured C40-single-phase ($\text{Mo}_{0.85}\text{Nb}_{0.15}$) Si_2 crystal in the 0°- and 45°-orientations as functions of the applied stress. For comparison purposes, the SSCR of MoSi_2 single crystals along the [001] hard orientation, which was reported by Inui et al. [20], is included.

Fig. 11. Expected temperature dependencies of the yield stresses for the three C11_b variants and C40 phase in the lamellar-structured crystals deformed in the (a) 45°- and (b) 0°-orientations. For comparison purposes, the yield (fracture) stress of the lamellar-structured crystal at 1400 °C is included.

Fig. 12. The temperature corrected SSCR versus stress curves of lamellar-structured $(\text{Mo}_{0.85}\text{Nb}_{0.15})\text{Si}_2$ crystals crept in the (a) 45°- and (b) 0°-orientations, respectively. These figures show that the creep data obtained in the present study can be modeled by a single constitutive equation (4) shown in the text.

Fig. 13. Microstructures and phase-volume fractions of the (a, b) non-added ternary and (c, d) Cr-added lamellar-structured crystals annealed at 1400 °C for 168 h. (a, c) Crystal orientation maps for the constituent C11_b phases. The observation direction was parallel to $[10\bar{1}0]$, but the corresponding crystal orientation maps were colored along a direction rotated towards $[11\bar{2}0]$ by 15° from $[10\bar{1}0]$ to distinguish the orientation colors of the three variant C11_b phases. (b, d) Volume fractions of the constituent phases in the crystals. The gray segments represent effective strengthening phases.

Fig. A.1 SAED patterns from the C40 phase and C11_b variants of V1, V2 and V3 defined by Eqs. (1)–(3) taken along two different directions of (a) [10 $\bar{1}$ 0]_{C40} and (b) [11 $\bar{2}$ 0]_{C40} [7]. The observed direction was tilted by 30° around the [0001] axis each other in the C40 phase, as schematically shown in Fig. (c), to definitely distinguish them one another. (d) Crystallographic orientation relationships between the C40-matrix phase on (0001) and three variants (V1, V2, and V3) of the C11_b phase on (110) in the duplex-phase, lamellar-structured crystals.

Table A.1 Schmid factors for the possible operative slip systems in the C40-matrix phase and three C11_b variants constituting the duplex-phase crystal in the 0°- and 45°-orientations.

Slip System	0°-orientation				45°-orientation			
	Variant-type in C11 _b			C40	Variant-type in C11 _b			C40
	V1	V2	V3		V1	V2	V3	
Loading orientation	[001]	[33̄1̄]	[3̄3̄1̄]	[101̄0]	[1.735 1.735 1]	[6.465 0.465 1̄]	[0.465 6.465 1̄]	[1 0 1̄ 1.228]
{110}<11̄1]	0	0.375	0.375	—	0.433	0.433	0.433	—
{100}<010]	0	0.375	0.375	—	0.250	0.062	0.062	—
{101}<010]	0	0.463	0.463	—	0.365	0.183	0.183	—
{023}<100]	0	0.480	0.480	—	0.398	0.226	0.226	—
{013}<33̄1̄]	0.387	0.387	0.387	—	0.305	0.376	0.376	—
(0001)<112̄0>	—	—	—	0	—	—	—	0.433

Table A.2 Visible/invisible criteria for dislocations whose operation is expected in C11_b-V1 in the g·b contrast analysis shown in Fig. 4(a) and Fig.6.

phase	C11 _b -V1						
figure	Fig.4(a)	Fig.4(b)	Fig.4(c)	Fig.4(d)	Fig.6(a)	Fig.6(b)	Fig.6(c)
beam direction	$[\bar{1}01]$	$[\bar{1}01]$	$[\bar{3}3\bar{1}]$	$[\bar{3}3\bar{1}]$	$[\bar{1}11]$	$[\bar{3}31]$	$[\bar{1}\bar{1}1]$
Burgers g-vector vector	006	110	$10\bar{3}$	$0\bar{1}\bar{3}$	110	$1\bar{1}6$	$1\bar{1}2$
[100]	invisible	visible	visible	invisible	visible	visible	visible
[010]	invisible	visible	invisible	visible	visible	visible	visible
[110]	invisible	visible	visible	visible	visible	invisible	invisible
[1 $\bar{1}$ 0]	invisible	invisible	visible	visible	invisible	visible	visible
[111]	visible	visible	visible	visible	visible	visible	visible
[11 $\bar{1}$]	visible	visible	visible	visible	visible	visible	visible
[1 $\bar{1}$ 1]	visible	invisible	visible	visible	invisible	visible	visible
[1 $\bar{1}$ $\bar{1}$]	visible	invisible	visible	visible	invisible	visible	invisible
[331]	visible	visible	invisible	visible	visible	visible	visible
[33 $\bar{1}$]	visible	visible	visible	invisible	visible	visible	visible
[$\bar{3}$ 31]	visible	invisible	invisible	invisible	invisible	visible	visible
[$\bar{3}$ 3 $\bar{1}$]	visible	invisible	visible	visible	invisible	invisible	visible

Table A.3 Visible/invisible criteria for dislocations whose operation is expected in C11_b-V2 in the g·b contrast analysis shown in Fig. 4(b) and Fig.6.

phase	C11 _b -V2						
figure	Fig.4(f)	Fig.4(g)	Fig.4(h)	Fig.4(i)	Fig.6(a)	Fig.6(b)	Fig.6(c)
beam direction	$[1\bar{1}1]$	$[1\bar{1}1]$	$[1\bar{1}1]$	$[001]$	$[1\bar{1}0]$	$[3\bar{3}1]$	$[1\bar{1}0]$
<div>g-vector Burgers vector</div>	$\bar{1}01$	$0\bar{1}\bar{1}$	110	$1\bar{1}0$	110	$1\bar{1}\bar{6}$	$00\bar{4}$
$[100]$	visible	invisible	visible	visible	visible	visible	invisible
$[010]$	invisible	visible	visible	visible	visible	visible	invisible
$[110]$	visible	visible	visible	invisible	visible	invisible	invisible
$[1\bar{1}0]$	visible	visible	invisible	visible	invisible	visible	invisible
$[111]$	invisible	visible	visible	invisible	visible	visible	visible
$[11\bar{1}]$	visible	invisible	visible	invisible	visible	visible	visible
$[1\bar{1}1]$	invisible	invisible	invisible	visible	invisible	visible	visible
$[1\bar{1}\bar{1}]$	visible	visible	invisible	visible	invisible	visible	visible
$[331]$	visible	visible	visible	invisible	visible	visible	visible
$[33\bar{1}]$	visible	visible	visible	invisible	visible	visible	visible
$[3\bar{3}1]$	visible	visible	invisible	visible	invisible	invisible	visible
$[3\bar{3}\bar{1}]$	visible	visible	invisible	visible	invisible	visible	visible